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Manufacturing Methods and Technology Engineering

for

"Growth of Large Diameter Nd:YAG

Laser Crystals"

by

R. Uhrin and R.F. Belt

Interim Progress Report
April 1, 1980 to March 31, 1981
Contract No. DAAB 07-77-C-0375
Modification P00003

Airtron Division
Litton Industries, Inc.
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Morris Plains, N.J. 07950

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Continued experimentation on the growth of Nd:YAG in boule sizes of 2 inch diameter was performed in this report period. The axial and radial gradients received more attention. It was found that the use of a Dy_2O_3 stabilized zirconia for insulation has given a very favorable growth gradient for our 4.5 x 4.5 inch crucibles. Twelve runs were started over a year. The first five of these were poor quality due to growth or apparatus difficul-

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered) ties. The next runs showed steady improvements which culminated in several good boules close to 2 inch diameter. These had few cracks, blossoms, or other defects. The first delivery of 12 engineering laser rods was taken from one completed boule. The tested rods were found to meet all specifications for the program. At least one boule has been obtained which should yield more than 30 rods.

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A

The objective of this program is to investigate methods for increasing the boule diameter of Nd:YAG production growth runs without deterioration of laser rod quality. A goal of two inches would nearly double present rod yields.

PURPOSE

The production growth of Nd:YAG for laser rods is performed exclusively by the Czochralski method. These boules are currently 1.25 - 1.50 inches in diameter. The purpose of this program is to obtain a larger yield of high quality rods by increasing the diameter of the grown boule. Preliminary investigation has shown that a goal of 2.0 inches could nearly double rod yields for many Army requirements.

This program consists of several parts including crystal growth, rod fabrication, and passive testing for quality. Laser rods provided under the program are required to meet existing military specifications for AN/GV5-5 applications.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

FOREWORD

This interim technical report on Manufacturing Methods and Technology Engineering for "Growth of Large Diameter Nd:YAG Laser Rods" was prepared by the Airtron Division of Litton Industries, Inc., Morris Plains, New Jersey 07950 under Contract No. DAAB 07-77-C-0375 for the Solid State and Injection Laser Team of the U.S. Army Electronics Research and Development Command, Night Vision and Electro Optics Laboratory, Fort Belvoir, Virginia 22060.

The program was initiated by Mr. John Strozyk. During the first year it was monitored by Mr. William Comeyne. The present monitor is Ms. Kay Chloupek. The production of Nd:YAG for laser rods is performed exclusively by the Czochralski method. These boules are currently 1.25 - 1.50 inch in diameter. The purpose of this program is to obtain a larger yield of high quality rods by increasing the diameter of the grown boule. Preliminary investigation has shown that a suitable goal of 2.0 inches could nearly double rod yield for small sizes. The same growth equipment and procedures are utilized.

This project consists of several parts including crystal growth, rod fabrication, and passive testing for quality. Laser rods provided under the program are required to meet existing military specifications for a (4.3 x 43)mm rod used in the GVS-5 and other Army programs.

1.0 Introduction

Growth of Nd:YAG crystals of larger diameter than is normally available from current production processes has distinct advantages for the laser industry. Improvements in laser rod yield per unit time cannot be expected from increased growth rate since this has already been maximized. Therefore, the alternative is to increase the crystal diameter in production in order to meet the increased industrial demand for laser rods.

Solving this problem is not a simple matter of scaling up current production systems. The high growth temperature and attendant power requirements demand larger and more efficient power supplies just to satisfy an increase in crucible size. More important than this is the crystal size increase which puts constraints on the growth process that aren't encountered at this time in production. This is specifically associated with increased strain in the crystals.

The first six months of work on this program concentrated on working with the larger growth systems and is contained in an earlier report[1]. During that portion of the program it was possible to solve most of the problems associated with the new systems and techniques required to handle the large growth system. The main growth difficulty at that time was cracking prior to the crystal reaching final diameter. Thus the main effort during the current portion of the program has been to determine a technique for getting the crystal to final diameter and obtaining a crystal of good quality. Both of these objectives were met at the end of the reporting period.

2.0 Experimental

The basic approach utilized to achieve good quality growth has been to adjust the crucible position within the coil as a means of varying the radial temperature gradient in the melt. It is not clear what the correct gradient should be with the type of growth station design employed. However previous growth results indicate that a steeper radial gradient is required based on the severity of crystalline defects which have appeared prior to the crystal reaching final diameter. In all growth experiments the radial temperature gradient has been 25-30°C per centimeter. This is substantially lower than the existing gradient in production growth stations and has been a difficult parameter to control.

2.1 Growth Station Design

A conceptual view of the basic crystal growth station is presented in Figure 1. A 4.5 inch diameter and 4.5 inch high iridium crucible with cover (A) is supported by concentric zirconia tubes (B). This arrangement is surrounded by zirconia grain insulation (C) which is enclosed by a quartz glass tube (D). Power is supplied to the crucible

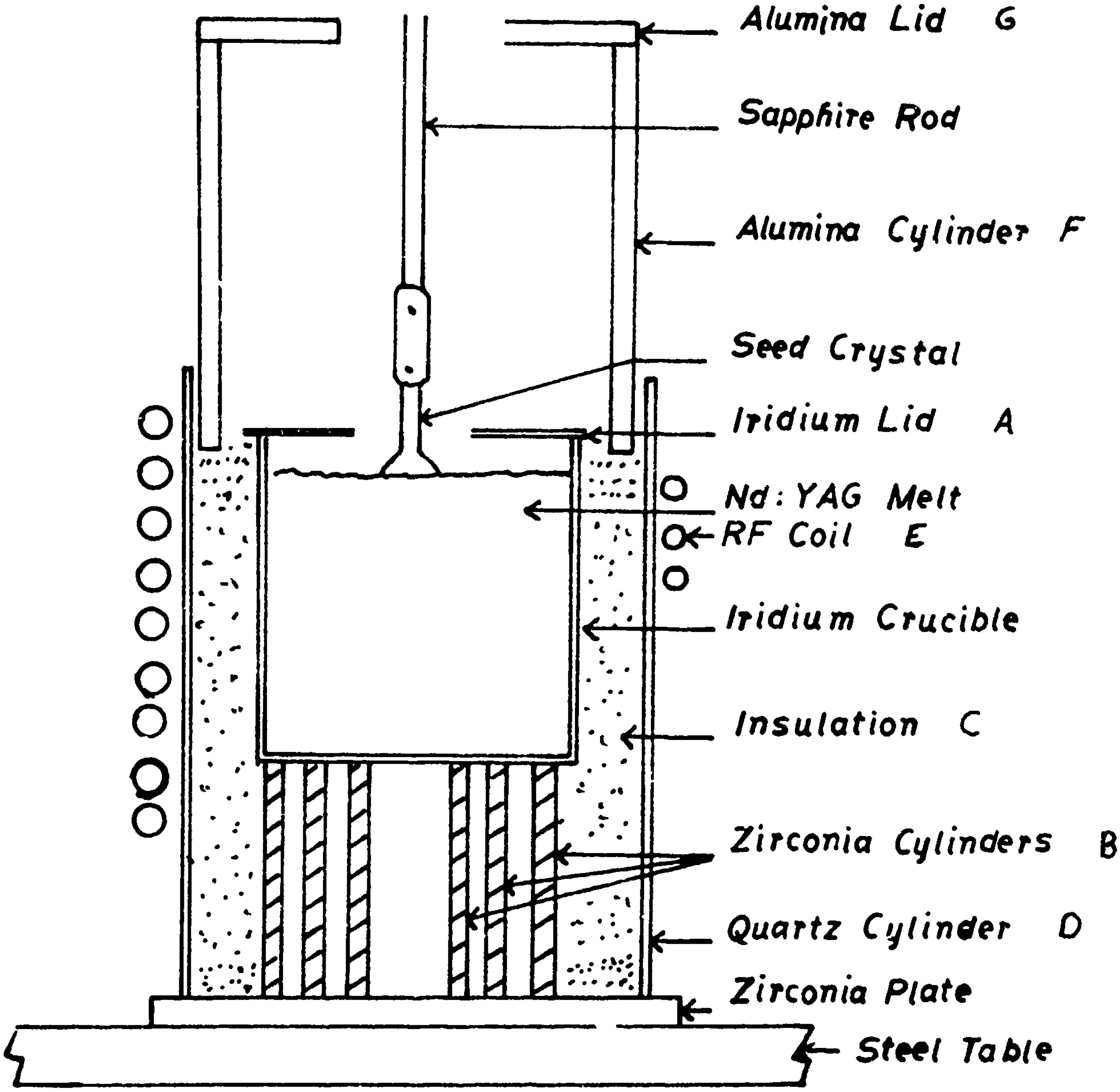

Figure 1 Drawing of Basic Growth Station

by means of an rf coil constructed from circular copper tubing (E). The area above the crucible into which the crystal is pulled is insulated by means of an alumina tube (F) and an alumina cover (G). Figure 2 shows the arrangement.

2.2 Gradient Control

The radial melt temperature gradient is controlled to a great degree by the insulating capability and amount of the insulation surrounding the crucible. However, the amount of insulation is limited by the requirement of intimate coupling of the crucible with the rf field. Thus the available radial gradient is somewhat fixed by melt properties and then outside design constraints. During the course of this experimental work it was possible to effect small gradient changes with the axial positioning of the crucible within the coil as depicted in Figure 1. Since the gradient is not completely linear as shown in the earlier report[1], this effect is quite subtle with respect to linearizing the gradient in the region of 1/2-1 inch of melt radius.

The axial temperature gradient within the growth station was not measured in the absence of an acceptable method of making the temperature measurements. However the configuration of the ceramic components was designed according to that utilized under production conditions. This did not appear to favor cracking in the absence of internal crystalline defects. In fact later in the program full length

Figure 2(a) Basic crucible and bottom insulation

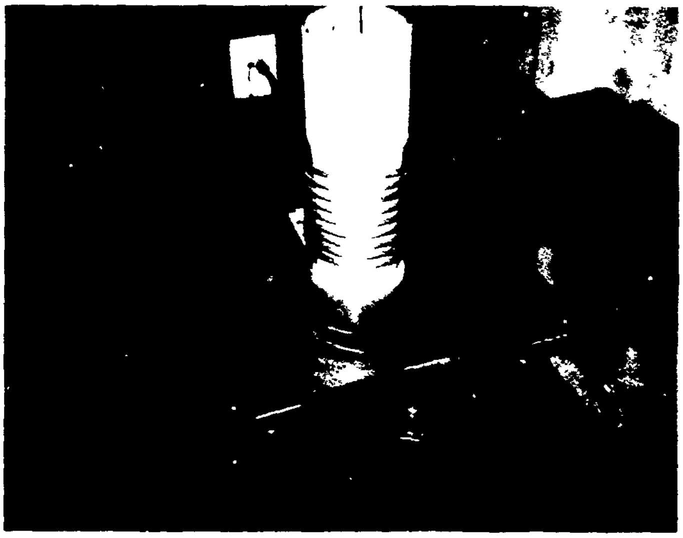

Figure 2(b) Top alumina tube and cover attached, seed rod lowered crystals with gross defects were obtained in an uncracked state.

3.0 Growth Run Results

Growth results at the beginning of this reporting period followed the trend existing at the end of the earlier period. It was possible to obtain flawless growth out to final diameter, but shortly after reaching diameter the crystal generally experienced an upset in the growth process which led to crystalline defects and subsequent cracking. Nevertheless steady progress was made on a run-to-run basis such that by the end of the reporting period it was possible to reliably obtain at least three inches of good quality growth at about two inch diameter.

3.1 Review of Growth Runs

Table I summarizes the growth runs completed since the end of the previous reporting period. All growth experiments were conducted utilizing 4.5 inch diameter and 4.5 inch high iridium crucibles with the water-cooled growth station enclosure.

An evaluation of the radial melt temperature gradient indicated that the available gradient was lower in the established growth station design than in production stations. Therefore it was felt that stable growth with the lower gradient could be obtained only at a lower growth rate. As a result, the first growth run (2349) in the period was

Table I

Summary of Crystal Growth Runs

Run #	Crystal Diameter	Pull Rate	Rotation Rate	Remarks
2349	1.5	0.0125	15	Lower pull rate and smaller diameter produced flawless crystal which cracked from blossom generated by power interruption toward end of run.
2363	N/A	N/A	N/A	No crystal obtained in several growth attempts. Crucible was found to be leaking.
2429	N/A	N/A	N/A	No crystal obtained in several growth attempts. Crucible was found to be leaking.
2435	1.75	0.018	15	Crystal melted off after 1 inch of straight growth due to power supply problem. No apparent crystal defect but crystal cracked from melt off.
2463	1.85	0.0125	15	Crystal melted off after 2 inch of straight growth. Power supply problems led to power loss and crystal cracked. Only 2 small defects in straight section.
2464	1.95	0.0125	15	Power loss after 2 inches of straight growth. Crystal cracked during quench. Poor diameter control. Large blossom after reaching diameter.
2484	1.95	0.0125	15	Crystal melted off after 2 inch of straight growth. Good diameter control. Crystal had large blossoms first before reaching diameter.

Table I

Summary of Crystal Growth Runs (Continued)

Run #	Crystal Diameter	Pull Rate	Rotation Rate	Remarks
2500	1.90	0.0125	15	Good diameter control but crystal quality was poor. Crystal cracked during cooling. Run proceeded to full extent.
2533	1.95	0.0125	15	Good diameter control Crystal had large blossom before reaching diameter and small blossoms late in run. Crystal yielded engineering sample of 12 rods.
2542	1.90	0.0125	15	Good diameter control but crystal of poor quality. Run proceeded to full extent and crystal did not crack during cooling.
2572	1.90	0.0125	15	Good diameter control. Crystal flaw-less except for blossom at end of growth cycle.
2649	1.90	0.0125	15	Good diameter control. Crystal was flawless for full length of growth.

made with a pull rate of 0.0125 inch per hour. Even though the crystal was grown at a smaller diameter than normal the growth results appear to support that conclusion. A power interruption toward the end of the growth run generated a blossom which led to cracking. However, the crystal was otherwise free of defects. No defects were present prior to the crystal reaching final diameter or shortly thereafter.

The next two growth runs were not completed because of leaks which developed in the crucible wall. However the following experiment (2435) was performed at a slightly larger diameter and at the customarily higher growth rate. This crystal melted off after a short section of growth at diameter due to a power supply problem. Since there were no evident crystalline defects prior to this occurrence this result would tend to dispute the lower growth rate argument. Nevertheless the next growth run (2463) was made at the lower growth rate and in spite of another power supply problem the crystal had only two small defects in the at-diameter section.

Beginning with run 2464 a significant improvement was observed in the crystal growth results. Although there were still problems with defect generation the general crystal quality improved to the point where extended lengths of growth at diameter were attained without cracking. Growth runs 2464 and 2484 virtually duplicated the previous experiment. In each case there was a large "blossom" generated as the crystals reached diameter and they cracked after reaching about two inches of growth at diameter. The following growth run (2500) was also of poor quality, but growth of this crystal proceeded to its full extent before the crystal cracked during the cooling process.

The remaining four growth runs all proceeded to completion without cracking and the first sample of laser rods was extracted from crystal 2533. This crystal contained defects on each side of the section from which the laser rods were obtained, but this central section was of good quality. Although the following growth run (2542) was of poor quality, the final two growth runs were of excellent quality and these crystals yielded the remaining required laser rods for the engineering and demonstration samples.

3.2 Problem Analysis

The greatest difficulty in this program has come from defect generation as the crystal approaches final diameter in its growth cycle. Although constitutional supercooling is inherent in the growth of Nd:YAG and slow growth rates must be utilized as a result, the aforementioned problem does not seem to be related mainly to growth rate. This becomes apparent when one considers the relatively slow growth rate utilized toward the end of the reported experimental work.

Data on the typical radial melt temperature gradient obtained with the growth station design used for this work was presented in the earlier report. The most significant aspect of this data is that the gradient does not change much with small changes in the growth station design. While it would be desirable to obtain a gradient as near as possible to that existing in production, this leads to some serious problems with the growth stations involved.

One important feature is that a gradient of the required magnitude would push the crucible wall temperature close to the iridium melting point. This would make operation at the growth temperature marginal and associated growth operations procedurally difficult. Another feature is that the additional heat loss from the growth station due to a higher radial gradient puts some constraints on the power supply in that the higher power output required raises the output voltage to a point where high voltage discharges tax the power supply's reliability. In spite of these difficulties a unique solution was found to modifying the radial melt temperature gradient. There appears to be a direct association between the institution of this change and the better growth results toward the end of the above experimental work.

It should be noted that most of the insulating ceramics utilized for crystal growth are composed of zirconia. While this material possesses good thermal insulating properties by virtue of its relatively low thermal conductivity, it is also quite transparent to blackbody radiation at the crystal growth temperature.

Figure 3 represents a curve of the blackbody radiation[2] for a body radiating at 2300°K, the approximate melting temperature of Nd:YAG. The peak of this curve lies at about 1.2 m where zirconia is transparent. Thus much of the infrared energy passes through the normal insulation used in the growth station. A material doped with Dy^{3+} has very strong absorption at this same wavelength. (See Figure 4) A novel approach was utilized in order to effectively limit the amount of radiation escaping from the growth station and thus improve the insulating properties.

An alternate type of zirconia insulation was prepared by crystallizing the cubic form of ZrO_2 stabilized with Dy_2O_3 rather than Y_2O_3 or CaO which are normally used. This material was prepared by growing Dy_2O_3 (40 mole percent) stabilized cubic zirconia crystals with a patented growth process and then reducing these crystals to a granular form compatible with the growth station design. This procedure was initiated with growth run N2533 where the top two inches of insulation surrounding the crucible were replaced with the alternate insulation. A pyrometric probe of the radial melt temperature gradient for this run showed what appeared to be a refinement of the gradient near the center of the melt. While this was a gross measurement the true effect was realized

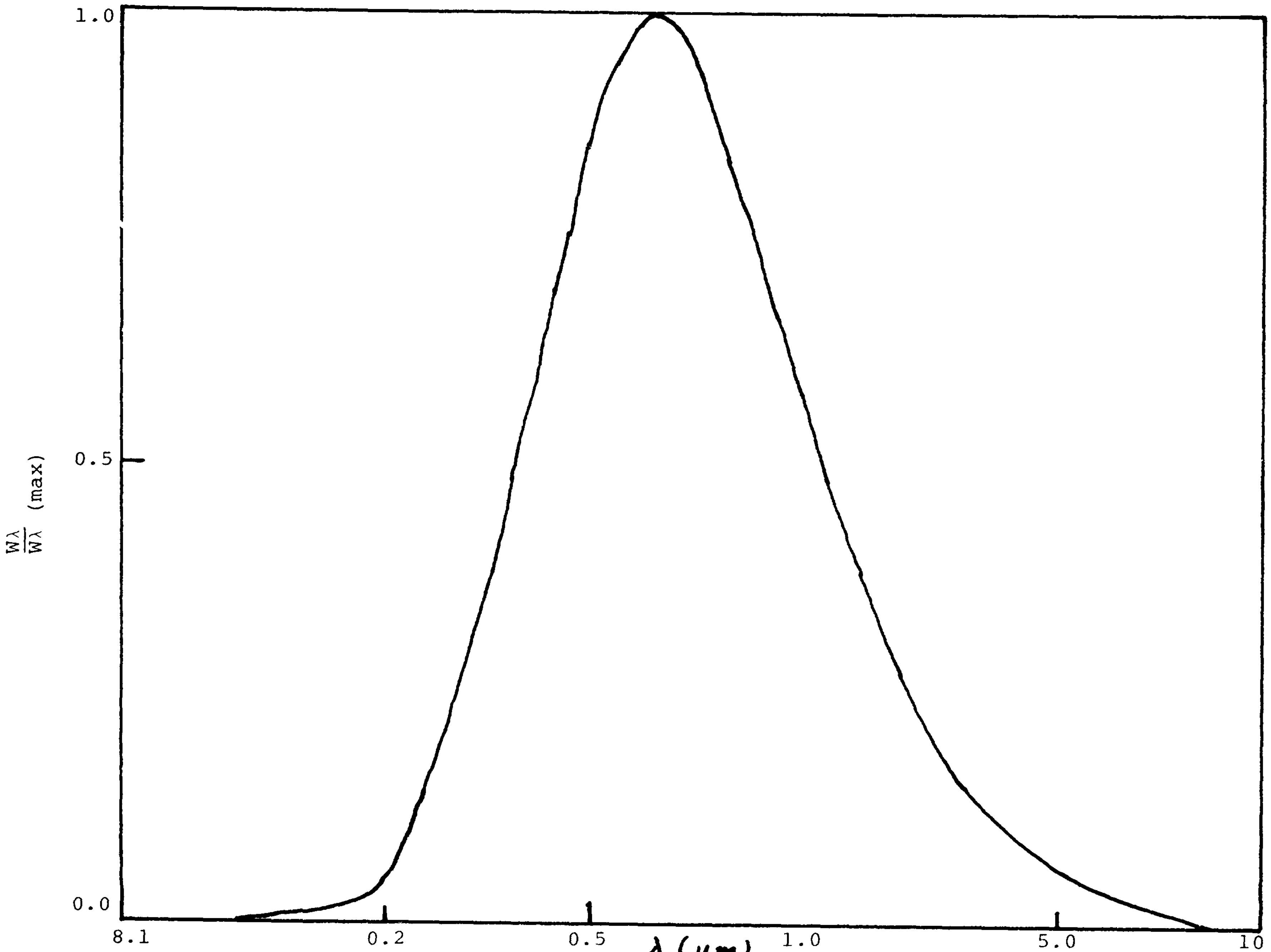

Figure 3 Emission from a Blackbody at 2300K

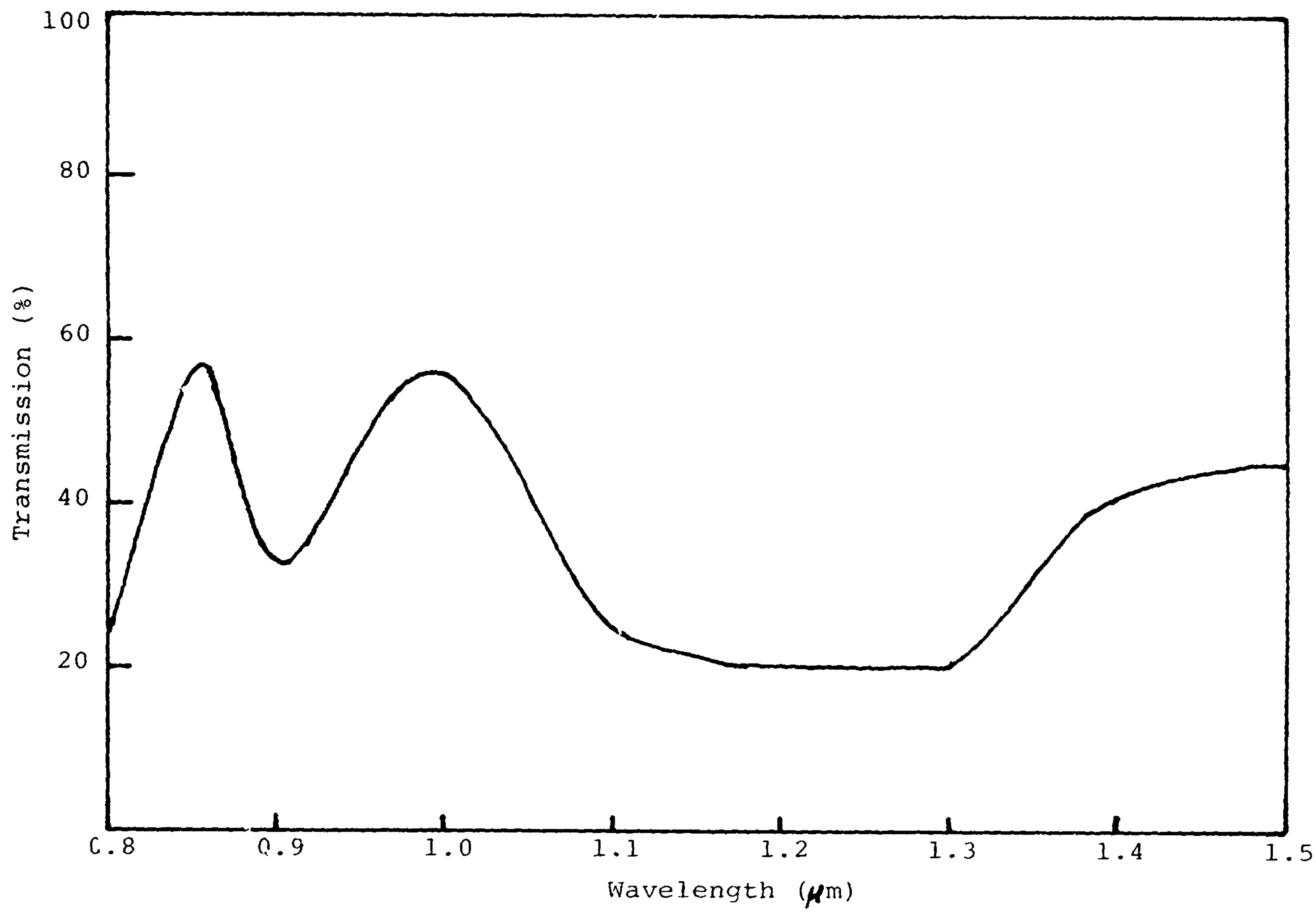

Figure 4 Absorption of Dy_2O_3 Stabilized Zirconia

when this crystal growth cycle was brought to completion without the crystal cracking. This was the first time a growth run at the large diameter was brought to completion in such a manner. The three subsequent runs also were completed by replacing all of the insulation surrounding the crucible with this alternate material. The absence of cracking in spite of extensive flaws (N-2542) indicated lower bulk crystal strain using this approach.

The real effect of this design change is not completely understood at this point but it is theorized that instability in the melt convection has been eliminated near the melt top center. Thus the tendency for generation of defects at the crystal core has been reduced. The next problem which has to be dealt with is some further modification of the growth interface to offset the tendency for defect generation due to constitutional supercooling. This has been approached initially by reducing the growth rate. However, this is an undesirable situation for improving the growth efficiency. It is believed at this time that the thermal convection in the absence of crystal growth is quite similar to standard production crystal growth. Further refinement in the growth can be expected, therfore, by evaluating the effects of crystal rotation rates on the growth quality and this will be approached in future work.

3.3 Preparation of Laser Rods for Engineering Sample Delivery

The first delivery of this contract was prepared during this interim report period. It consisted of twelve engineering sample laser rods which were to be obtained from a single Nd:YAG boule. These rods were not required to meet the proposed specifications in their entirety. However for reference purposes we reproduce in Table II an abbreviated set of the contract laser rod specifications. These were derived from U.S. Army SCS 507 Documents plus selected amendments to the present program. Essentially these rods conform to the AN/GVS-5 type rod specification.

The boule from which the laser rods were extracted was N-2533 which was listed in Table I. This boule was one of the first to give good growth results. It contained only a few blossoms in the taper region and several near the end of growth. Otherwise the quality was reasonably good. A picture of the boule is given in Figure 5 for reference. More than 12 rods were core drilled from the rough boule. However the best rods (as judged by optical and visual testing) were selected for complete fabrication. The fabrication was performed in the Airtron laser rod shop by regular personnel using techniques and procedures in daily use. Table III is a summary of the test data which were accumulated during and after the fabrication. These data meet the proposed specification of Table II

Table II

Laser Rod Specifications from
SCS 507 and Amendments

Characteristics	Specification	Test Method
Nd Dopant	1.0 - 1.3 atomic %	Fluorescent Lifetime ($220 \pm 15 \mu s$)
Dimension	Length 43.0 mm $^{+2}_{-0}$ mm	Calipers
	Dia. 4.27 $\pm$ 0.02mm	Micrometer
End Surface Quality	20 - 5	Comparison Standards
End Surface Flatness	$\lambda/5$	Optical Flat
Parallelism	10 sec	Fizeau Interferometer
Perpendicularity	5 min	Autocollimator
Strain	<0.5 Fringe/43mm	Twyman Green - Double Pass
End Coating	End 1: 60 $\pm$ 3% R	Cary Spectrophotometer
	End 2: AR with <0,25% loss	

Table III

Summary of Test Data for Twelve Engineering Sample Laser Rods from Boule N-2533

Rod Number	Diam. (mm)	Length (mm)	Perpen. (min)	Parallelism (sec)	Lifetime (μs)	Scatt. Sites	Strain (Total fringe)
R4071	4.27	45.8	2.5	<10	206	2	<0.1
R4072	4.27	45.8	2.0	<10	210	10	<0.1
R4073	4.26	45.8	2.5	<10	201	10	0.1
R4074	4.27	45.7	2.5	<10	218	20	0.2
R4075	4.26	45.8	2.5	<10	210	10	0.2
R4076	4.27	45.8	0.5	<10	208	5	<0.1
R4077	4.28	45.9	2.0	<10	215	5	<0.1
R4079	4.27	45.8	2.0	<10	219	<5	<0.1
R4080	4.27	44.3	1.5	<10	212	2	<0.1
R4081	4.28	45.8	1.5	<10	206	1	0.4
R4082	4.27	45.6	1.0	<10	214	<5	0.2
R4083	4.27	45.9	2.0	<10	204	<5	0.3

in every category. A few of the rods contained slight scattering sites of submicrometer size. The sites did not affect the total strain as exhibited by the Twyman-Green fringe pattern. Thus there should be little or no effect on the active operation. It appears that all of our passive tests indicate that rod quality is fully equal to that obtained from smaller diameter boules.

3.4 Description of Crystals

In order to give the reader an idea of the progress made under this program, we present some photographs of the actual crystals grown and described in Section 3.1. The first runs of Table I were plagued with cracking, blossoms, and other defects which rendered most of the crystals useless. No rods were obtained from about seven boules. The change of insulation material gave an improved yield almost immediately. Several crystals were obtained at nearly two inch diameter with no cracks. The first of these was Boule N2533 which is pictured in Figure 5(a). This boule had several blossoms near the top and bottom. However a length of 50 or more mm was grown in the center which was free of any defects. It was from this section that the 12 engineering samples were extracted. The tests of Section 3.3 showed that the quality of the material was excellent and all specifications could be met. The next boule was also complete and is pictured in Figure 5(b). The internal quality was not the best and the blossom location

Figure 5(a) View of Boule N2533
Between Crossed Polarizers

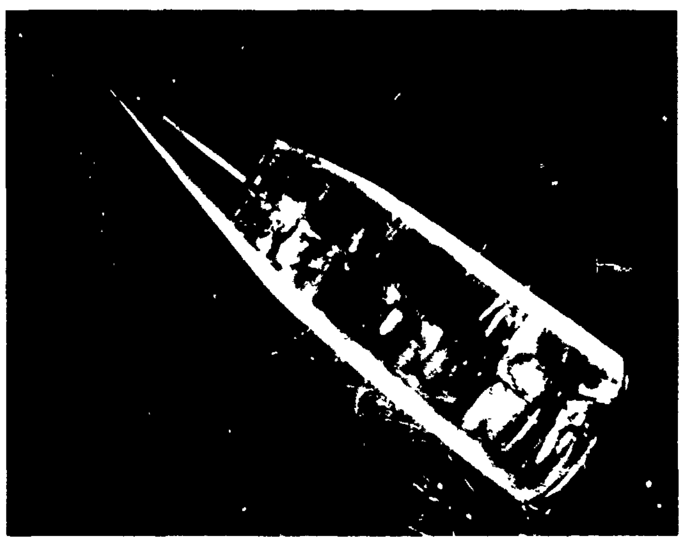

Figure 5(b) View of Boule N2542
Between Crossed Polarizers

prevented the extraction of good laser rods in any quantity.

The last two boules which were grown gave very good results and were nearly free of any major defects. The boule from run N2572 is pictured in Figure 6(a). This boule has been evaluated, core drilled, and rods fabricated. A total of 37 rods was obtained from the boule. These rods are now being evaluated for delivery as the confirmation run. Twelve of the best will be selected for the delivery. Essentially this boule meets all the growth and delivery requirements of the contract. Figure 6(b) shows a picture of the last boule grown in this report period. A full evaluation of quality has not been made but at least 25 - 30 rods can be obtained from the material. The rods are going to be core drilled and evaluated soon and results will appear in our final report.

At this stage it appears that a satisfactory production process has been defined. It remains to check this with a few additional growth runs at the 2 inch diameter. The principal difficulties appear to be related to the RF generator. The unit used for our 4.5 inch crucibles is operating at near capacity. Thus some changes may have to be made in equipment in order to guarantee a high probability of success.

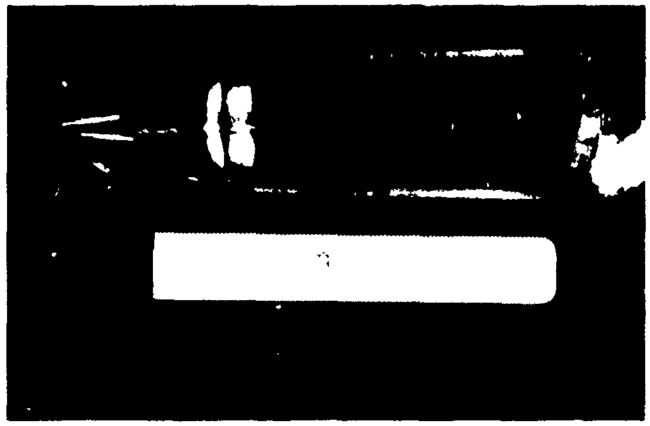

Figure 6(a) Boule from Run N2572. Ordinary Light. A yield of 37 rods was obtained.

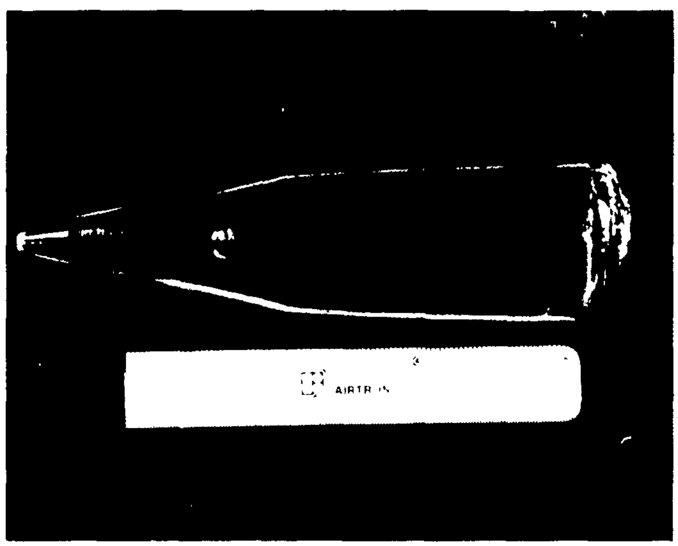

Figure 6(b) Boule from Run N2649. Ordinary Light.

4.0 Conclusions

Results of the most recent growth runs indicate that slow but steady progress has been made toward reaching a goal of two inch diameter by three inch length of high quality crystal. Initial growth difficulties prevented completion of any growth runs without cracking of the crystals. This was tied to the presence of severe blossom formations and extensive strain in the crystals. Experience in the production growth of Nd:YAG indicates that these effects are associated with instabilities due to an improper interface shape (not convex enough). Indeed, a reduction in crystal diameter and pull rate resulted in improved crystal quality (Runs 2349-2463) from these changes which were intended to lengthen the solid-liquid interface. Increased diameter (Runs 2464-2500) led to a degradation in crystal quality indicating that a further refinement in the growth interface shape was still required.

Assuming that the growth interface should match the shape of the melt isotherms the two possiblities are that the isotherms are either too steep or too shallow in relation to the growth interface. Considering the size of the growth system it is reasonable to believe that the radial melt temperature gradient is too large. Consequently a reduction in the infrared heat loss through use of the specially prepared insulation (Runs 2533-2649) provided a further improvement in crystal quality. An engineering sample of 12 laser rods was obtained from Run 2533 and either of two subsequent runs provided material for a confirmatory sample of 12 rods.

Since not much more can be done to better insulate the growth station and since the shape of the solid-liquid interface is fixed by the growth and rotation rates, it would appear the future work will have to be concerned with optimizing the rotation rate. It is felt that the melt isotherms are still too steep in relation to the growth interface and the direction should be toward lower rotation rates. Further reductions in the growth rate are unreasonable for efficient production.

5.0 Program for Next Period

Future growth experiments will continue to employ the specially prepared insulation in the growth station. Due to the improved results of recent growth runs it is anticipated that few if any other refinements will be made in the growth station design. Closer scrutiny will be given to optimizing the crystal rotation rate and increasing the growth rate where possible to make the growth process more efficient.

The deliveries of the confirmatory laser rods and the pilot production lot will be made. All other contract items will be completed to fulfill the original objectives.

6.0 References

1. R. Uhrin and R.F. Belt, Manufacturing Methods and Technology Engineering for "Growth of Large Diameter Nd:YAG Laser Crystals", Semiannual Progress Report, Oct. 1, 1979 to April 1, 1980, Contract No. DAAB07-77-C-0375 Mod P0003, July, 1980.

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Chief of Research and Development Department of the Army Army Research Office Washington, D.C. 20310	1
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Commander US Army Materiel Development & Readiness Command ATTN: DRCPP-IF (Mr. Byrnes) 5001 Eisenhower Avenue Alexandria, VA 22333	1
Division of Non-Ionizing Radiation Litterman Army Institute of Research Presidio of San Francisco San Francisco, CA 94129	1
Director US Army Industrial Base Engineering Activity ATTN: ARXIB-MT (Mr. C. E. McBurney) Rock Island Arsenal Rock Island, IL 61201	1
Dr. Fred W. Quelle Office of Naval Research 495 Summer Street Boston, Massachusetts 02210	1
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